

10/018765

JC10 Rec'd PCT/PTO 17 DEC 2001

CERTIFICATION UNDER 37 CFR 1.10

I hereby certify that this Transmittal Letter and the papers indicated as being transmitted therewith are being deposited with the United States Postal Service on this date shown below in an envelope as "Express Mail Post Office to Addressee" under the below indicated Mailing Label Number, addressed to: Box PCT, Commissioner for Patents, Washington, D.C. 20231.

Mailing Label No.: EF232848399USDeposit Date: December 17, 2001Shari Saus
Name: Shari SausATTORNEY'S DOCKET NO. TURKP0118US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
(DO/EO/US)

In re national phase of:

Applicant(s): Stephan Appelt, Giovanni D'Orsaneo, Nadim Joni Shah
International Application No.: PCT/EP00/05251
International Filing Date: June 7, 2000
Priority Date Claimed: June 18, 1999
Title of Invention: TEST CELL FOR A NOBLE GAS POLARIZER

**TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED
OFFICE (DO/EO/US) CONCERNING ENTRY INTO U.S. NATIONAL
PHASE UNDER 35 U.S.C. 371**

Box PCT
Commissioner for Patents
U.S. Patent and Trademark Office
Washington, D.C. 20231

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information under 35 U.S.C. 371:

1. This express request to immediately begin national examination procedures (35 U.S.C. 371(f)).
2. The U.S. National Fee (35 U.S.C. 371(c)(1)) and other fees (37 CFR 1.492) as indicated below.

3. A copy of the International application (35 U.S.C. 371(c)(2)):
 - a. ☒ is transmitted herewith
(International Publication No. WO 00/79244).
 - b. ☐ is not required, as the application was filed with the United States Receiving Office.
 - c. ☐ has been transmitted by the International Bureau. A copy of Form PCT/1B/308 is enclosed.
4. ☒ An accurate translation of the International application into the English language (35 U.S.C. 371(c)(2)) is transmitted herewith.
5. Amendments to the claims of the International application under PCT Article 19 (35 U.S.C. 371(c)(3)):
 - a. ☐ are transmitted herewith.
 - b. ☐ have been transmitted by the International Bureau.
6. ☐ An accurate translation of the amendments to the claims under PCT Article 19 (38 U.S.C. 371(c)(3)) is transmitted herewith.
7. A copy of the international preliminary examination report (PCT/IPEA/409)
 - a. ☐ is transmitted herewith.
 - b. ☐ is not required as the United States Patent and Trademark Office was the IPEA.
8. Annex(es) to the international preliminary examination report
 - a. ☐ is/are transmitted herewith.
 - b. ☐ is not required as the United States Patent and Trademark Office was the IPEA.
9. ☐ An accurate translation of the annexes to the international preliminary examination report is transmitted herewith.
10. ☐ An oath or declaration of the inventor (35 U.S.C. 371(c)(4)) complying with 35 U.S.C. 115 is submitted herewith.

11. An International Search Report (PCT/ISA/210)
- a. ☒ is transmitted herewith.
 - b. ☐ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was searched by the United States International Searching Authority.
12. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98 is transmitted herewith, along with Form PTO-1449 and copies of citations listed.
13. ☐ An assignment document is transmitted herewith for recording, along with a separate cover sheet.
14. ☒ A preliminary amendment is enclosed.
15. ☐ A verified statement claiming small entity status is enclosed.
16. ☐ Other:

Basic National Fee					Fee
IPEA - US					\$710.00
ISA - US					\$740.00
PTO not ISA or IPEA					\$1,040.00
Claims meet PCT Art. 33(1)-(4) - IPEA - US					\$100.00
Filing with EPO or JPO search report					\$890.00
Enter appropriate basic fee →					\$890.00
Claims*	Number filed		Number extra	Rate	
Total claims	12	-20	0	\$18.00	\$0.00
Independent claims	1	-3	0	\$84.00	\$0.00
Multiple dependent claims (if applicable)				\$280.00	
Total of above					\$890.00
Small entity statement enclosed, 1 if Yes, 0 if No →				0	\$0.00
Total national fee					\$890.00
Fee for recording enclosed assignment				\$40.00	
Total fees enclosed					\$890.00

*After any attached preliminary amendment reducing the number of claims and/or deleting multiple dependencies.

☒ [X] A check in the amount of \$ 890.00 to cover the above fees is enclosed.

☐ [] Please charge our Deposit Account No. 18-0988 in the amount of \$ _____. A duplicate copy of this sheet is enclosed.

WARNING: TO AVOID ABANDONMENT OF THE APPLICATION THE BASIC NATIONAL FEE MUST BE PAID WITHIN THE 20/30 MONTH TIME LIMIT.

10/018765

531 Rec'd PCT

17 DEC 2001

Transmittal Letter to United States Designated/Elected OfficePage 5

16. The Commissioner is hereby authorized to charge the following additional fees that may be required by this paper and during the entire pendency of this application to our Deposit Account No. 18-0988:

a. ☒ 37 CFR 1.492(a)(1), (2), (3), (4) and (5) (basic national fee)

WARNING: BECAUSE FAILURE TO PAY THE NATIONAL FEE WITHIN 30 MONTHS WITHOUT EXTENSION (37 CFR S 1.495(B)(2)) RESULTS IN ABANDONMENT OF THE APPLICATION, IT WOULD BE BEST TO ALWAYS CHECK THE ABOVE BOX.

b. ☐ 37 CFR 1.492(b), (c) and (d) (presentation of extra claims)

NOTE: Because additional fees for excess or multiple dependent claims not paid on filing or on later presentation must only be paid or these claims cancelled by amendment prior to the expiration of the time period set for response by the PTO in any notice of fee deficiency (37 CFR 1.492(d)), it might be best not to authorize the PTO to charge additional claim fees, except possibly when dealing with amendments after final action.

Respectfully submitted,



Don W. Bulson, Reg. No. 28,192

Direct all correspondence and telephone calls to:

Don W. Bulson, Esq.
RENNER, OTTO, BOISSELLE & SKLAR, P.L.L.
1621 Euclid Avenue, 19th Floor
Cleveland, Ohio 44115
Tel: 216-621-1113 Fax: 216-621-6165
Internet: dbulson@rennerotto.com

10011971057052802

10/018765

531 Rec'd PCT/F... 17 DEC 2001

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re National Phase of:

Applicant: Stephan Appelt et al.
PCT Application No.: PCT/EP00/05251
PCT Filing Date: June 7, 2000
Title: SAMPLE CELL FOR AN INERT GAS POLARISER
Attorney Docket No. TURKP0118US

PRELIMINARY AMENDMENT DELETING MULTIPLE DEPENDENCIES

Commissioner for Patents
United States Patent and Trademark Office
Washington, DC 20231

Sir:

Please amend the application in accordance with the following appended parts:

- A. Clean Version of Replacement Paragraph/Section/Claim with Instructions for Entry; and
- B. Version with Markings to Show Changes Made.

Remarks

By way of the foregoing, all of the claims have been amended to delete multiple dependencies. In the event there still remains a claim that depends from more than one claim, the Office is hereby authorized to amend such claim to depend from the first mentioned of the multiple claims from which it depends.

Respectfully submitted,



Don W. Bulson, Reg. No. 28,192
RENNER, OTTO, BOISSELLE & SKLAR, LLP
1621 Euclid Avenue - Nineteenth Floor
Cleveland, Ohio 44115
(216) 621-1113

A. Clean Version of Replacement Paragraph/Section/Claim
with Instructions for Entry

Please amend the application as follows:

In the Claims:

Please substitute the following claims for the pending claims of corresponding number.

3. Sample cell according to claim 1, characterised in that the sample cell comprises two plane windows (2, 3) which are arranged parallel to one another.
4. Sample cell according to claim 1, characterised by two projecting radii (8) of at least one millimetre on either side of the window or windows, which radii are arranged directly adjacent to the circumference of the window and form a pressure-resistant joint with the rim of the orifice.
5. Sample cell according to claim 1, characterised by borosilicate glass of which the glass body (1) is composed and borofloat glass of which the windows (2, 3) are composed.
6. Sample cell according to claim 1, characterised in that the glass (1, 2, 3) is at least 5 mm thick.
7. Sample cell according to claim 1, characterised in that the external diameter of the glass body (1) is between 20 and 100 millimetres, in particular between 35 and 40 millimetres.
8. Sample cell according to claim 1, characterised in that the sample cell is part of a polariser for polarising a inert gas.
9. Sample cell according to claim 1, characterised in that valves (6, 7) which are produced from glass and comprise ring seals made of ethylene propylene are provided at the entrance and exit for the gas.

10. Method of producing a sample cell according to claim 1, which is characterised in that a glass body with an orifice, an inlet and an outlet is provided, a plane window is inserted into the orifice, the circumference of the window corresponding to the circumference of the orifice and the rim of the orifice being thicker than the thickness of the window, and the rim is heated from the exterior at least twice in such a way that the glass in the vicinity of the rim melts, creating a fused joint between the window and the rim.

11. Method according to claim 10, in which the rim or rims of each window is ground prior to insertion and prior to fusion with the rim of the orifice of the glass body, in particular the rim is ground cylindrically and is then cleaned with an acid, in particular with hydrofluoric acid.

12. Use of a sample cell according to claim 1 at pressures of at least 10 bar.

B. Version with Markings to Show Changes Made

Please amend the application as follows:

In the Claims:

3. (Amended) Sample cell according to claim 1 [or 2], characterised in that the sample cell comprises two plane windows (2, 3) which are arranged parallel to one another.
4. (Amended) Sample cell according to [any of the preceding claims] claim 1, characterised by two projecting radii (8) of at least one millimetre on either side of the window or windows, which radii are arranged directly adjacent to the circumference of the window and form a pressure-resistant joint with the rim of the orifice.
5. (Amended) Sample cell according to [any of the preceding claims] claim 1, characterised by borosilicate glass of which the glass body (1) is composed and borofloate glass of which the windows (2, 3) are composed.
6. (Amended) Sample cell according to [any of the preceding claims] claim 1, characterised in that the glass (1, 2, 3) is at least 5 mm thick.
7. (Amended) Sample cell according to [any of the preceding claims] claim 1, characterised in that the external diameter of the glass body (1) is between 20 and 100 millimetres, in particular between 35 and 40 millimetres.
8. (Amended) Sample cell according to [any of the preceding claims] claim 1, characterised in that the sample cell is part of a polariser for polarising a inert gas.
9. (Amended) Sample cell according to [any of the preceding claims] claim 1, characterised in that valves (6, 7) which are produced from glass and comprise ring seals made of ethylene propylene are provided at the entrance and exit for the gas.
10. (Amended) Method of producing a sample cell according to [any of the preceding claims] claim 1, which is characterised in that a glass body with an orifice, an inlet and

an outlet is provided, a plane window is inserted into the orifice, the circumference of the window corresponding to the circumference of the orifice and the rim of the orifice being thicker than the thickness of the window, and the rim is heated from the exterior at least twice in such a way that the glass in the vicinity of the rim melts, creating a fused joint between the window and the rim.

11. (Amended) Method according to [the preceding claim] claim 10, in which the rim or rims of each window is ground prior to insertion and prior to fusion with the rim of the orifice of the glass body, in particular the rim is ground cylindrically and is then cleaned with an acid, in particular with hydrofluoric acid.

12. (Amended) Use of a sample cell according to [any of the apparatus claims] claim 1 at pressures of at least 10 bar.

1/PRTS

10018765.032802

10/018765

531 Rec'd PCT.

17 DEC 2001

Sample cell for an inert gas polariser

5 The invention relates to a sample cell for an inert gas polariser. The invention also relates to a method of producing the sample cell.

10 A sample cell for an inert gas polariser is known which consists of glass, a gas inlet and a gas outlet and through which the light of a laser is conveyed.

15 Recent developments in magnetic resonance tomography (MRT) and in magnetic resonance spectroscopy (NMR) with polarised inert gases have led to expectations of applications in medicine, physics and material sciences. High polarisation of nuclear spins may be achieved by optical pumping using alkali atoms, as demonstrated by the document Happer et al, Phys. Rev. A, 29, 3092 (1984). At present, rubidium is typically used in the presence of an inert gas and nitrogen. This enables nuclear spin polarisation of the inert gas xenon (^{129}Xe) of about 20 percent to be achieved. This nuclear spin polarisation is about 100,000 times greater than the equilibrium polarisation in clinical magnetic resonance tomographs. The associated drastic increase in the signal-to-noise ratio explains why possible new applications in medicine, science and technology are expected in future.

25 The term polarisation denotes the degree of orientation (ordering) of the spin of atomic nuclei or electrons. 100 percent polarisation denotes, for example, that all nuclei or electrons are oriented in the same manner. The polarisation of nuclei or electrons is associated with a magnetic moment.

30 Polarised xenon is, for example, inhaled by or injected into a person. The polarised xenon accumulates in the brain 10 to 15 seconds later. The distribution of the inert gas in the brain is detected by magnetic resonance tomography. The result is used for further analysis.

The choice of the inert gas depends on the application. ^{129}Xe has a great chemical shift. If xenon, for example, is adsorbed on a surface, its resonant frequency changes significantly. Xenon also dissolves in fat-loving (i. e. lipophilic) liquids. Xenon is used when these properties are desired.

5

The inert gas helium barely dissolves in liquids. Therefore, the isotope ^3He is generally used when cavities are concerned. The human lung is an example of such a cavity.

10

Some inert gases have valuable properties different from those mentioned above. For example, the isotopes ^{83}Kr , ^{21}Ne and ^{131}Xe have a quadrupole moment which is of interest, for example, for experiments in basic research and in surface physics. However, these inert gases are very expensive so they are unsuitable for applications demanding relatively large amounts.

15

It is known from the document B. Driehuys et al, Appl. Phys. Lett. 69, 1668 (1996) to polarise inert gases in the following manner.

20

Circularly polarised light, i. e. light in which the angular momentum or the spin of the photons all point in the same direction is prepared using a laser. The angular momentum of the photons is transmitted to free electrons of alkali atoms. The spins of the alkali atom electrons therefore deviate greatly from the thermal equilibrium. The alkali atoms are therefore polarised. The polarisation of the electron spin is transmitted from an alkali atom to an inert gas atom by a collision

25

between an alkali atom and an inert gas atom. Polarised inert gas is thus formed.

30

Alkali atoms are used as they have a great optical dipole moment which interacts with the light. Alkali atoms also have a respective free electron which prevents disadvantageous interactions between two and more electrons per atom.

Caesium would be a particularly suitable alkali atom and is superior to

rubidium for achieving the aforementioned effects. At present, however, there are no lasers available with sufficiently high power, as required for the polarisation of xenon by means of caesium. However, it is expected that lasers with powers of 100 watts at the wavelength of caesium will be developed in future. Then, caesium will probably preferably be used for the polarisation of inert gases.

In the prior art, a gaseous mixture is conveyed slowly under a pressure typically of 7 to 10 bar through a cylindrical glass cell. The gaseous mixture consists of 98 percent of ⁴helium, one percent of nitrogen and one percent of xenon. The typical velocities of the gaseous mixture are a few ccm per second.

The gaseous mixture initially flows through a vessel (hereafter called "storage vessel") in which there is about one gram of rubidium. The storage vessel with the rubidium located therein is heated together with the adjoining glass cell to about 100 to 150 degrees Celsius. The rubidium is vaporised by providing these temperatures. The concentration of the vaporised rubidium atoms in the gaseous phase is determined by the temperature in the storage vessel. The gas stream conveys the vaporised rubidium atoms from the storage vessel into the cylindrical sample cell. A powerful circularly polarised laser (100 watt power in continuous operation) passes axially through the sample cell (also hereinafter called "glass cell") and optically pumps the rubidium atoms into a highly polarised state. The wavelength of the laser must be adapted to the optical absorption line of the rubidium atoms (D1 line). In other words, for optimum transmission of the polarisation of the light to an alkali atom, the frequency of the light must coincide with the resonant frequency of the optical crossover. The sample cell is located in a static magnetic field of a few 10 gauss which is generated by a coil (pair of Helmholtz coils). The direction of the magnetic field extends parallel to the cylinder axis of the sample cell or parallel to the beam direction of the laser. The magnetic field serves to guide the polarised alkali atoms. The rubidium atoms which are optically highly polarised by the light of the laser collide in the glass cell, inter alia with the xenon atoms, and transmit their high polarisation to the xenon atoms. On leaving the sample cell, the rubidium settles on the wall owing to the

high melting point in comparison to the melting points of the other gases. The polarised xenon or the gaseous mixture is relayed from the sample cell into a freezeout unit. This consists of a glass flask of which the end is immersed in liquid nitrogen. The glass flask is also located in a magnetic field with a strength of 1,000 to 2,000 gauss. The highly polarised xenon gas settles on the internal glass wall of the freeze-out unit as ice when the glass wall is immersed into the liquid consisting of nitrogen. At the outlet of the freeze-out unit, the remaining gas (helium and nitrogen) is conveyed via a needle valve and finally discharged.

The flow rate in the entire arrangement can be controlled by the needle valve and can be measured using a measuring device. If there is an excessive increase in the flow rate, there is no time for transmission of polarisation from the rubidium atoms to the xenon atoms, so no polarisation is achieved. If the flow rate is too low, too much time elapses before the desired amount of highly polarised xenon freezes. In fact polarisation of the xenon atoms decreases again due to relaxation. Relaxation of the xenon atoms is markedly decelerated by the freezing and by the strong magnetic field to which the freeze-out unit is exposed. It is therefore necessary to freeze the inert gas as quickly as possible and with a minimum of loss after polarisation. Although relaxation cannot be avoided owing to freezing, there are still 1 to 2 hours at $T=77\text{ K}$ before polarisation has diminished to such an extent that further use of the initially highly polarised gas is no longer possible.

A certain amount of energy is required to polarise a single free alkali atom. The energy required corresponds to the resonant frequency for raising the free electron of the alkali atom from the basic state into an excited state. For optimum transmission of the energy from a laser to the alkali atom, the frequency of the laser light must be adapted to the resonant frequency of the alkali atom. Lasers generally transmit their light within a specific frequency spectrum. This is not a single frequency but a distribution of frequencies. The available laser spectrum is characterised by the so-called line width. Alkali atoms are polarised most effectively if the line width of the laser coincides with the line width of the optical

crossover of the alkali atom. The optical line width of an alkali atom is accordingly proportional to the pressure of the added ⁴helium laser (pressure broadening). In fact, the higher the pressure, the higher the number of collisions between an alkali atom and a collision partner such as helium.

5

The 100 watt strong laser used in the prior art is a glass fibre-coupled diode laser with a typical spectral width of 2 nanometres. At a gas pressure of 10 bar, the line width of the optical crossover of rubidium atoms is broadened to about 0.3 nanometres. Therefore, only a fraction of the laser light is used in existing rubidium/xenon polarisers in which expensive diode lasers with a typical line width of 2 nanometers are used for optical pumping. If the much cheaper diode lasers with a line width of 4 to 5 nm are used at 10 bar gas pressure, the efficiency is still much lower.

10

15

In a gaseous mixture according to the prior art, the partial pressures of helium are currently up to 10 bar. This is very high in comparison with the other partial pressures. In addition to the pressure broadening of the optical crossover of the alkali atom, the high partial pressure means that polarised atoms only rarely reach the sample wall of the glass cell and lose their polarisation there, for example due to interaction with paramagnetic centres. The likelihood that polarised atoms will disadvantageously collide with the cell wall decreases with increasing partial pressure.

20

25

In order to utilise the full power of the laser and at the same time to reduce disadvantageous relaxation effects due to collisions with the wall, helium pressure far above 30 bar would have to be employed.

30

The following should also be noted when putting together the gaseous mixture.

After absorbing a photon originating from the laser, an alkali atom such as rubidium is capable of emitting a photon due to fluorescence (fluorescence

photon). If this fluorescence photon is captured by an adjacent polarised alkali atom, this capture leads to the depolarisation of the alkali atom. The nitrogen, used in the polarisation of inert gases, in the gaseous mixture suppresses ("quenches") this fluorescence radiation in order to reduce the aforementioned undesirable depolarisation. The nitrogen partial pressure is typically 0.1 bar in the prior art.

The heavy inert gas atoms, for example xenon atoms, cause marked relaxation in the polarisation of the alkali atoms during collisions with the alkali atoms. To maximise the polarisation of the alkali atoms during optical pumping, the partial pressure of the inert gases in the gaseous mixture has to be suitably low. Even with a xenon partial pressure in the gaseous mixture of only 0.1 bar, laser powers of 100 watts are required to achieve 70 percent polarisation of the alkali atoms in the entire sample volume.

Glass cells blown from one piece of glass are used in the prior art. In the past, there was no other way to produce a glass cell which was capable of withstanding the desired high pressures and at the same time ensured high optical quality. With the aforementioned production of the glass cell, the windows through which the laser light enters and issues are invariably curved or rounded. Undesirable disadvantageous lens effects occur during the ingress or egress of the laser light. The laser light is focussed or expanded. This considerably impairs the efficiency with which alkali atoms in the gaseous mixture of the glass cell are polarised.

A glass cell for the polarisation of inert gases is to satisfy the following requirements.

It must withstand a high pressure of at least 10 bar and be non-magnetic and resistant to alkali metals at temperatures of up to 200 degrees Celsius.

The glass cell should be adapted to be closed by valves. The valve heads

or ring seals must withstand 200 degree Celsius in the presence of the gaseous mixture and must also be nonmagnetic and pressure-resistant. The influence of the valves on the polarisation of the inert gas should be as low as possible.

5 The surface in the interior of the cell should not have a destructive influence on xenon or rubidium polarisation. Therefore, there should be no paramagnetic or even ferromagnetic centres on the internal wall of the cell. The material making up the cell should be absolutely non-magnetic.

10 The laser light should be adapted to be propagated through the cell as far as possible without lens effects, i. e. in parallel.

15 The entry window of the cell should absorb as little as possible of the laser light. Otherwise, in particular the entry window will be excessively heated and ultimately destroyed.

20 The entry window should not be birefringent either at normal pressure or at high pressure. Otherwise, the circular polarisation of the laser would be destroyed or at least diminished.

25 An object of the invention is to provide a sample cell which is superior to the prior art in meeting these requirements.

30 The object of the invention is achieved by a glass cell having the features of the first claim. A method of producing the glass cell comprises the features of the independent claim. Advantageous developments emerge from the sub-claims.

35 The glass cell according to the claims comprises a glass body with an orifice. A plane glass window is inserted into the orifice. The shape and size of the window corresponds to the shape and size of the orifice. The orifice comprises a rim which is wider than the thickness of the window. This is to allow extensive contact between the window and the rim of the orifice. The rim of the window is

fused with the rim of the orifice.

A glass cell comprising a flat entry window can thus be provided. This allows a parallel beam path through the window.

5

The glass body also comprises a gas inlet and a gas outlet.

10

The glass body has, in particular, the form of a cylinder. The plane window is then formed by a disc. The diameter of the disc corresponds to the internal diameter of the glass body. The disc is inserted a short distance into the cylinder. The internal wall of the cylinder then provides a large contact area which can be fused with the rim of the disc. This internal face then forms the rim of the orifice which is broader than the thickness of the window.

15

Advantageously, the two orifices of the cylinder are sealed by a respective plane window.

The cylinder further comprises a gas inlet and a gas outlet.

20

In order to join the window rigidly to the glass cell, the rim is heated several times, in particular to 1,400 to 1,500 degrees Celsius so the respective region is melted several times. If the glass body is a cylinder, it is heated from the exterior in order to fuse an inserted window with the cylinder.

25

A radius of 1 to 2 mm is created at the internal and external edge of the window owing to the repeated melting. As a result, the glass cell is pressure-resistant and at the same time has at least one plane entry window.

30

Advantageously, a window is ground prior to fitting and prior to fusion with the rim of the orifice of the glass body and is then cleaned with an acid, in particular with 15 percent hydrofluoric acid. In particular, the rim is ground cylindrically. These steps ensure a quite particularly durable joint with the rim of

the orifice of the glass body.

Borosilicate glass is provided as material for the glass body or borofloat glass (borosilicate glass 3.3 ISO standard) for the window or windows. Both materials have the same coefficient of thermal expansion and may therefore be fused particularly well. Furthermore, both borosilicate glasses have few paramagnetic centres and are virtually not birefringent when pressure is applied, for example like quartz glass.

The glass body, for example the cylinder, typically has an internal diameter of 24 millimetres. The wall thickness is 5 millimetres or greater. A radius which joins the window to the glass body typically projects by 1 to 2 millimetres.

A glass cell of this type withstands a pressure of 15 bar.

Valves produced predominantly from glass are advantageously provided at the entrance and at the exit of the sample cell. If ring seals have been inserted for the glass valves, they consist of ethylene propylene. Ethylene propylene is resistant to alkalis and has no paramagnetic centres. Furthermore, the material absorbs virtually no inert gas.

No significant loss of polarisation was found immediately before and after this valve after NMR measurement of xenon polarisation.

The invention will be explained in more detail with reference to the following figures.

Fig. 1 shows the basic construction of the sample cell. The sample cell is composed of a cylinder 1 consisting of glass and two plane windows 2 and 3. The plane windows 2 and 3 are inserted a short distance into the respective ends of the cylinder. The windows 2 and 3 are rigidly joined to the cylinder 1. The gaseous mixture is initially conveyed into the sample cell and out again after

polarisation. The tubes 4 and 5 may be sealed by valves 6 and 7. Inlet tube 4 has a portion of enlarged diameter between the valve 6 and the sample cell 1, 2, 3. This portion receives alkali metal which vaporises here and can then be transported into the sample cell.

5

10

Fig. 2 is a cross-section of the sample cell. Entry window 2 and exit window 3 are inserted a short distance into the cylinder 1. The windows 2 and 3 have been joined to the cylinder 1 by repeated melting. The radii 8 form a fused joint between the plane windows 2 and 3 and the cylinder 1. The radii 8 are located on either side of a respective window 2 and 3 and adjoin the cylinder 1. The cylinder 1 consists of borosilicate glass. The windows consist of borofloat glass. The cylinder wall is 5 mm thick. The windows also have a wall thickness of 5 millimetres. The external diameter of the cylinder is about 35 to 40 mm.

15

Borofloat glass and borosilicate glass have the same coefficients of thermal expansion. The windows therefore fuse particularly well with the cylinder. A very pressure-resistant cell then exists, which even withstands pressures of 15 bar.

20

25

Fig. 3 is a cross-section through a valve 6 or 7. The end of a line 4 (tube) consisting of glass opens into a funnel 9. A thread 10 adjoins the funnel. A cap 11 composed of plastics material is screwed onto the thread 10. The cap 11 has a corresponding internal thread. A bolt 12 consisting of glass is fastened centrally internally on the cap 11. The bolt 12 penetrates into the funnel 9. The bolt 12 comprises two grooves containing O-rings 13. The O-rings 13 consist of ethylenepropylene. A branching tube located at the upper rim with the large diameter of the funnel 9 is not shown. An intermediate length of pipe, not shown, from which a tube branches may also be provided between funnel 9 and thread 10.

30

Once the cap 11 is screwed in, the leading O-ring produces a tight seal with the funnel 9. The valve is then closed.

The bolt 12 can have a greater external diameter than the internal diameter of the tube 4 and, at the end facing the funnel, a tapering portion which opens into the funnel. This portion acts as a counterpart to the funnel. An O-ring is provided at the tapering portion in such a way that the tapering portion ends tight against the funnel 9 when the bolt 12 has been moved sufficiently far in the direction of the funnel by screwing of the cap 11. The aforementioned high pressures are possible, in particular, with this embodiment.

Claims

1. Sample cell with a glass body (1) comprising a gas inlet (4) and a gas outlet (5) and at least one orifice,

-a plane glass window (2) is inserted into the orifice,

-the shape and size of the window (2) corresponds to the shape and size of the orifice,

-the orifice is limited by a rim,

-the rim is wider than the thickness of the window,

-the rim of the window is fused with the rim of the orifice.

2. Sample cell according to claim 1, characterised in that the glass body has the form of a cylinder (1), the plane window (2) being formed by a disc which is inserted into the glass body at one end of the cylinder.

3. Sample cell according to claim 1 or 2, characterised in that the sample cell comprises two plane windows (2, 3) which are arranged parallel to one another.

4. Sample cell according to any of the preceding claims, characterised by two projecting radii (8) of at least one millimetre on either side of the window or windows, which radii are arranged directly adjacent to the circumference of the window and form a pressure-resistant joint with the rim of the orifice.

5. Sample cell according to any of the preceding claims, characterised by borosilicate glass of which the glass body (1) is composed and borofloat glass of which the windows (2, 3) are composed.

6. Sample cell according to any of the preceding claims, characterised in that the glass (1, 2, 3) is at least 5 mm thick.

7. Sample cell according to any of the preceding claims, characterised in that

the external diameter of the glass body (1) is between 20 and 100 millimetres, in particular between 35 and 40 millimetres.

8. Sample cell according to any of the preceding claims, characterised in that the sample cell is part of a polariser for polarising an inert gas.

9. Sample cell according to any of the preceding claims, characterised in that valves (6, 7) which are produced from glass and comprise ring seals made of ethylene propylene are provided at the entrance and exit for the gas.

10. Method of producing a sample cell according to any of the preceding claims, which is characterised in that a glass body with an orifice, an inlet and an outlet is provided, a plane window is inserted into the orifice, the circumference of the window corresponding to the circumference of the orifice and the rim of the orifice being thicker than the thickness of the window, and the rim is heated from the exterior at least twice in such a way that the glass in the vicinity of the rim melts, creating a fused joint between the window and the rim.

11. Method according to the preceding claim, in which the rim or rims of each window is ground prior to insertion and prior to fusion with the rim of the orifice of the glass body, in particular the rim is ground cylindrically and is then cleaned with an acid, in particular with hydrofluoric acid.

12. Use of a sample cell according to any of the apparatus claims at pressures of at least 10 bar.

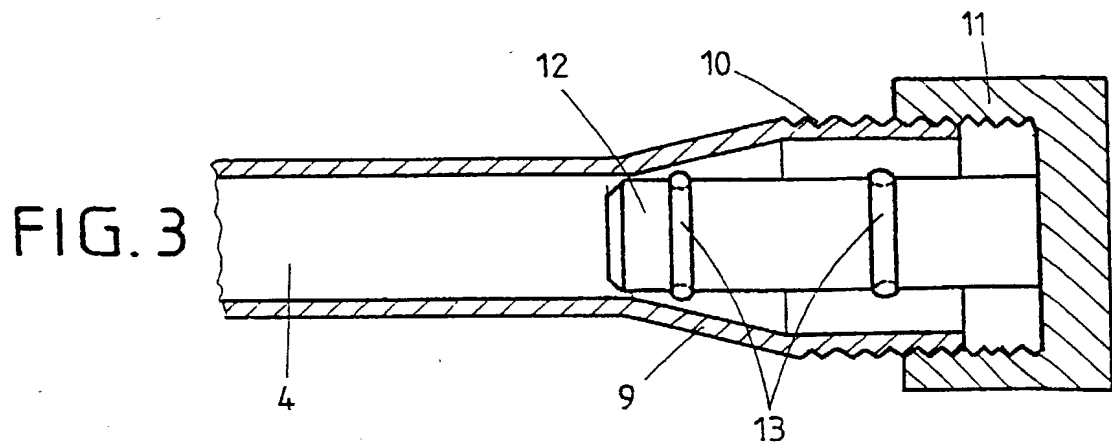
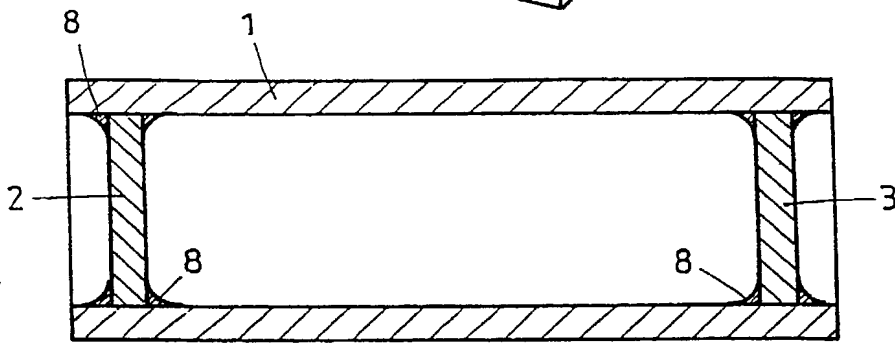
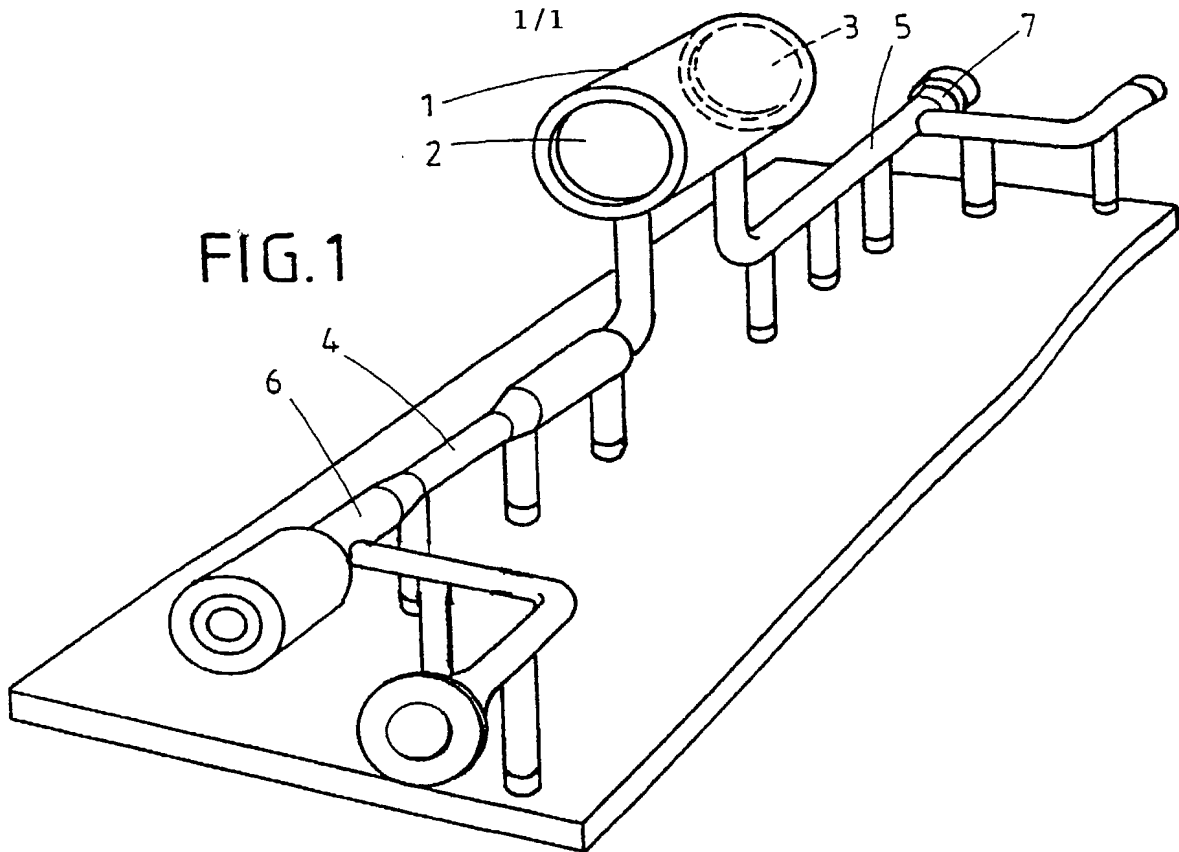
Abstract

5 The invention relates to a sample cell with a glass body comprising a gas inlet and a gas outlet and at least one orifice, a plane glass window is fused into the orifice, the shape and size of the window corresponds to the shape and size of the orifice, the orifice is limited by a rim, the rim is wider than the thickness of the window, the rim of the window is fused with the rim of the orifice.

10 The invention further relates to a method of producing the sample cell. The window is inserted into the orifice. The rim of the orifice is heated several times from the exterior in such a way that the glass melts and a fused joint is formed between the window and the rim of the orifice.

15 The sample cell produced by the method has a particularly rigid joint between the window and the glass body. Therefore, this glass cell is able to withstand pressures above 10 bar and allows the passage of light without lens effects owing to its plane-parallel windows.

20 The sample cell is used in a polariser for inert gas.



RENNER, OTTO, BOISSELLE & SKLAR

Attorney Docket No. TURKP0118US

PATENT (OUS)

COMBINED DECLARATION AND POWER OF ATTORNEY
(ORIGINAL, DESIGN, NATIONAL STAGE OF PCT)

As a below named inventor, I hereby declare that my residence, post office address and citizenship are as stated below next to my name; and I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

Title: SAMPLE CELL FOR AN INERT GAS POLARISER

the specification of which

☐ is attached hereto, or

☒ was filed as United States Application or PCT International Application (give Express Mail label number and deposit date if Application number not yet known):

Application No.: PCT/EP00/05251
(Express Mail Label No.)
Filing Date: December 18, 2001
(Deposit Date)
Amended on (if applicable):

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations §1.56(a).

PRIORITY CLAIM

I hereby claim priority benefits under Title 35, United States Code, §119 of (i) any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed; and (ii) any United States provisional application(s) that is/are listed below.

☐ no such applications have been filed.
☒ such applications have been filed as follows.

**EARLIEST FOREIGN/PROVISIONAL APPLICATION(S), IF ANY FILED WITHIN 12 MONTHS
(6 MONTHS FOR DESIGN) PRIOR TO THIS U.S. APPLICATION**

COUNTRY	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED	
			Yes	No
DE	199 27 788.5	18 June 1999	X	

**ALL FOREIGN APPLICATION(S), IF ANY FILED MORE THAN 12 MONTHS
(6 MONTHS FOR DESIGN) PRIOR TO THIS U.S. APPLICATION**

RENNER, OTTO, BOISSELLE & SKLAR

POWER OF ATTORNEY

As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (List name and registration number)

Armand P. Boisselle, Reg. No. 22,381; Warren A. Sklar, Reg. No. 26,373; Don W. Bulson, Reg. No. 28,192

The undersigned to this declaration and power of attorney hereby authorizes the U.S. attorney(s) named herein to accept and follow instructions from

Authorized representative: Gille Hrabal Struck Neidlein Prop Roos
Brucknerstraße 20
D-405-93 Düsseldorf, Germany

as to any actions to be taken in the Patent and Trademark Office regarding this application without direct communication between the U.S. attorney(s) and the undersigned. In the event of a change in the person(s) from whom instructions may be taken, the U.S. attorney(s) will be so notified by the undersigned.

Send Correspondence To

Don W. Bulson, Esq.
Renner, Otto, Boisselle & Sklar, P.L.L.
1621 Euclid Ave., 19th Floor
Cleveland, Ohio 44115

Direct Telephone Calls To:

(name and telephone number)

Don W. Bulson
(216) 621-1113

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

1-00

Full Name of Sole or First Inventor:		Stephan Appelt	
Inventor's signature:	Stephan Appelt	Date:	5.12.2001
Residence: (City & State/Country):	Same as Post Office address	Citizenship:	German
Post Office Address:	Gut Kartaus, Dürenerstraße 25 D-52428 Jülich, Germany DEX		

2-00

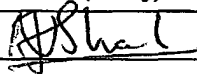
Full Name of Additional Joint Inventor (if any):		Giovanni D'Orsaneo	
Inventor's signature:	Giovanni D'Orsaneo	Date:	5.12.2001
Residence: (City & State/Country):	Same as Post Office address	Citizenship:	German
Post Office Address:	Artilleriestraße 72 D-52428 Jülich, Germany DEX		

CHECK FOR ANY OF THE FOLLOWING ADDED PAGE(S) WHICH
FORM A PART OF THIS DECLARATION

- [X] Signature for additional joint inventors.
[] Added page to combined declaration and power of attorney for divisional, continuation, or continuation-in-part (CIP) application.
[] This declaration ends with this page.

RENNER, OTTO, BOISSELLE & SKLAR

ADDED PAGE TO COMBINED DECLARATION AND POWER OF ATTORNEY
FOR SIGNATURE BY THIRD AND SUBSEQUENT INVENTORS

Full Name of Additional Joint Inventor (if any): Nadim Joni Shah			
Inventor's signature: 		Date:	11.01.02
Residence: (City & State/Country): Same as Post Office address		Citizenship:	German British
Post Office Address:		Vikarierstraße 4 Peter-Koof-Str. 8 D-52334 Linnich-Boslar, Germany D-52428 Juelich, Germany	

Full Name of Additional Joint Inventor (if any):			
Inventor's signature:		Date:	
Residence: (City & State/Country):		Citizenship:	
Post Office Address:			

Full Name of Additional Joint Inventor (if any):			
Inventor's signature:		Date:	
Residence: (City & State/Country):		Citizenship:	
Post Office Address:			

Full Name of Additional Joint Inventor (if any):			
Inventor's signature:		Date:	
Residence: (City & State/Country):		Citizenship:	
Post Office Address:			

Full Name of Additional Joint Inventor (if any):			
Inventor's signature:		Date:	
Residence: (City & State/Country):		Citizenship:	
Post Office Address:			